Recovery strategies for mirrors with boron carbide-based coatings for 6.x nm lithography

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Presentation overview

- Motivation for mirror recovery strategies
- Facilities at LLNL
- Optical constants of B and B_4C
- Survey of candidate mirror recovery techniques
- Work towards recovery of B_4C-coated mirrors for the LCLS FEL
  - The LCLS facility and its mirrors
  - LCLS coating lifetime issues
  - First experimental results
- Implications for 6.x nm lithography mirrors

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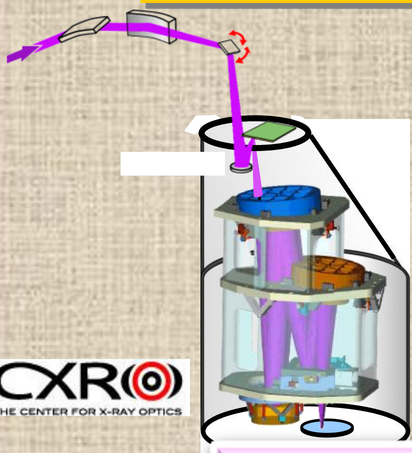
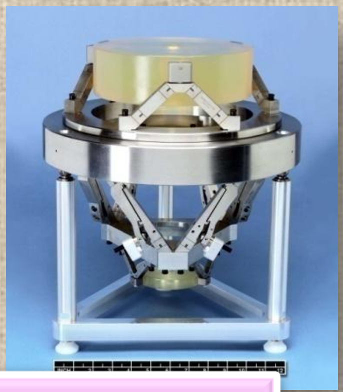
All optics will eventually be affected by contamination issues, especially when EUV wavelengths are involved

ETS



MET

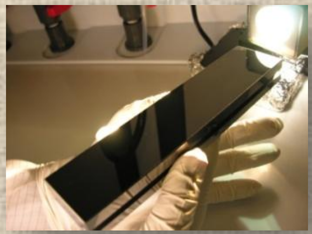


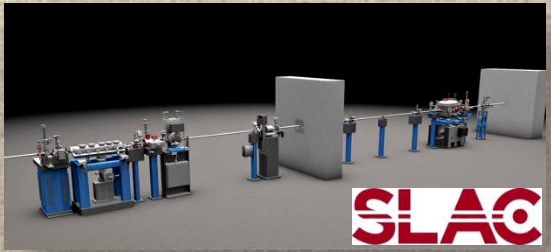
R. Soufli *et al*., *Proc. SPIE* 4343, 51 (2001)
R. Soufli *et al*., *Appl. Opt.* 46, 3736 (2007)

NASA's Solar Dynamics Observatory (SDO)

R. Soufli, *et al.*, Appl. Opt. 46, 3156-3163 (2007)
R. Soufli, *et al.*, Proc. SPIE 5901, 59010M (2005)
P. Boerner *et al*, Solar Physics 275, 41-66 (2012).
J. R. Lemen *et al*, Solar Physics 275, 17-40 (2012).

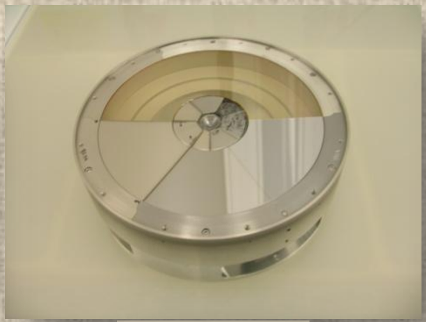
SXR beamline (LCLS)

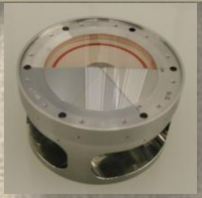




R. Soufli, M. Fernandez-Perea, *et al.*, Appl. Opt. 51, 2118 (2012)

NASA/NOAA's GOES-R space weather satellite





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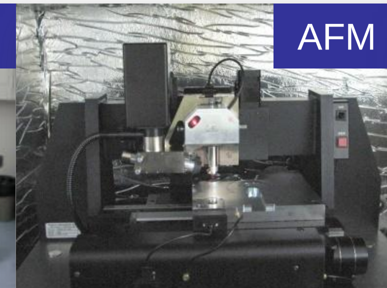
LLNL experimental facilities

DC- and RF-sputtering multilayer deposition systems

Precision surface metrology



Also (not pictured):

- Contact profilometers
- Thin film stress measurement apparatus

Custom cleaning facility for optical substrates

X-Ray Diffractometer

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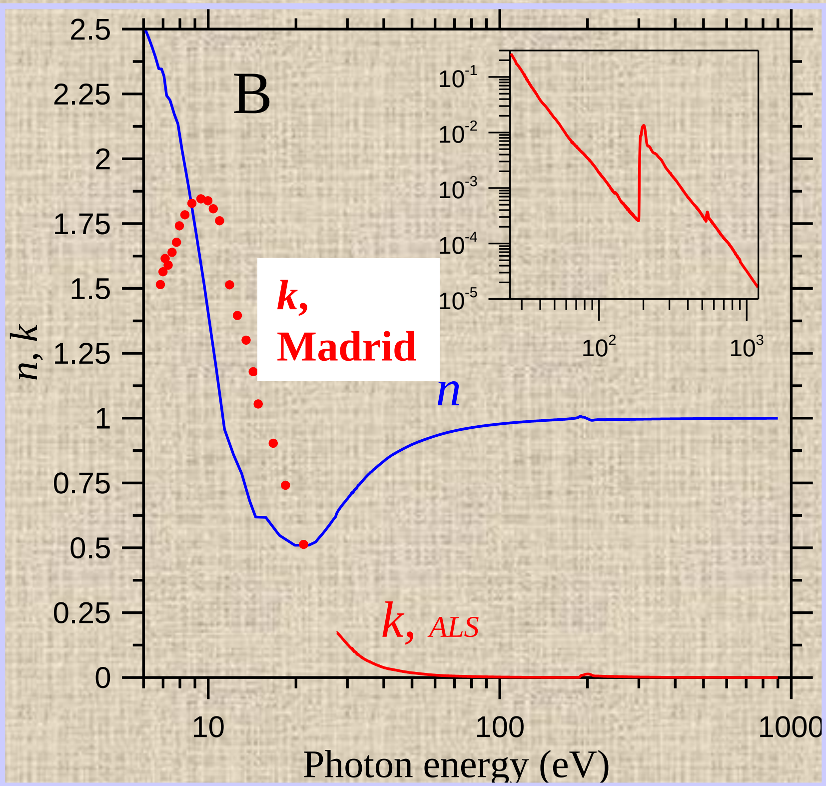
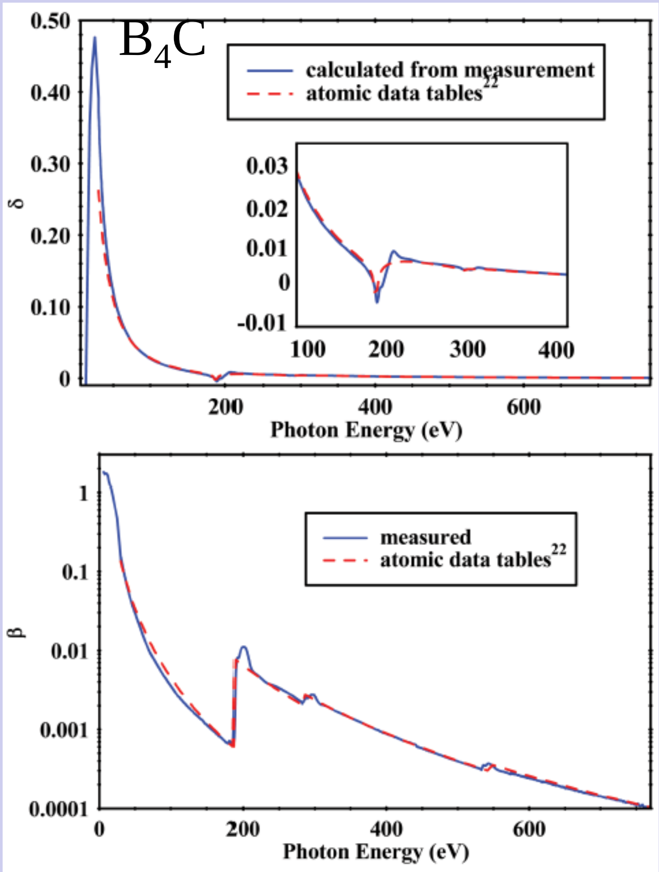
B and B_4C optical constants

- We have been involved in EUV-soft x-ray optical constants characterizations (transmittance + Kramers-Kronig formalism) of a wide variety of materials, including 6.x nm lithography candidates B and B_4C.
- Our B and B_4C optical constants have been experimentally confirmed with the results on La/B_4C, La_2O_3/B_4C (Yuriy Platonov) and LaN/B, LaN/B_4C (Eric Louis) multilayers presented in previous EUVL workshops.

$$N = n + ik = 1 - \delta + i\beta$$



M. Fernandez-Perea et. al., J. Opt. Soc. Am. A, Vol. 24 (2007)



R. Soufli et. al., Appl. Opt. 47, 4633 (2008)

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Candidate mirror recovery techniques

Coating recovery

- Usually work by forming volatile carbon species
- Favored because they preserve coating and can be implemented in situ

- **UV-ozone cleaning (currently used for synchrotron mirrors)**
- **Atomic oxygen/hydrogen plasma cleaning**

Requisites

Preserve coating
- **reflectance**
- **damage threshold**
- **thickness uniformity**

Avoid
- **material growth (e.g. oxides)**
- **coating erosion**
- **roughening**
- **chemical reaction**

Challenge: when C is a constituent of both the blemish and the coating

Substrate recovery

- Remove both contamination and coating

- **UV-ozone (for B_4C and B, in development)**
- **Release layers**
- **Chemical etching**

Requisites

Preserve substrate
- **roughness**
- **figure**
- **composition**

Avoid
- **material growth (e.g. oxides)**
- **substrate erosion**
- **chemical reactions**

Disadvantage: Needs re-coating, not possible in situ.

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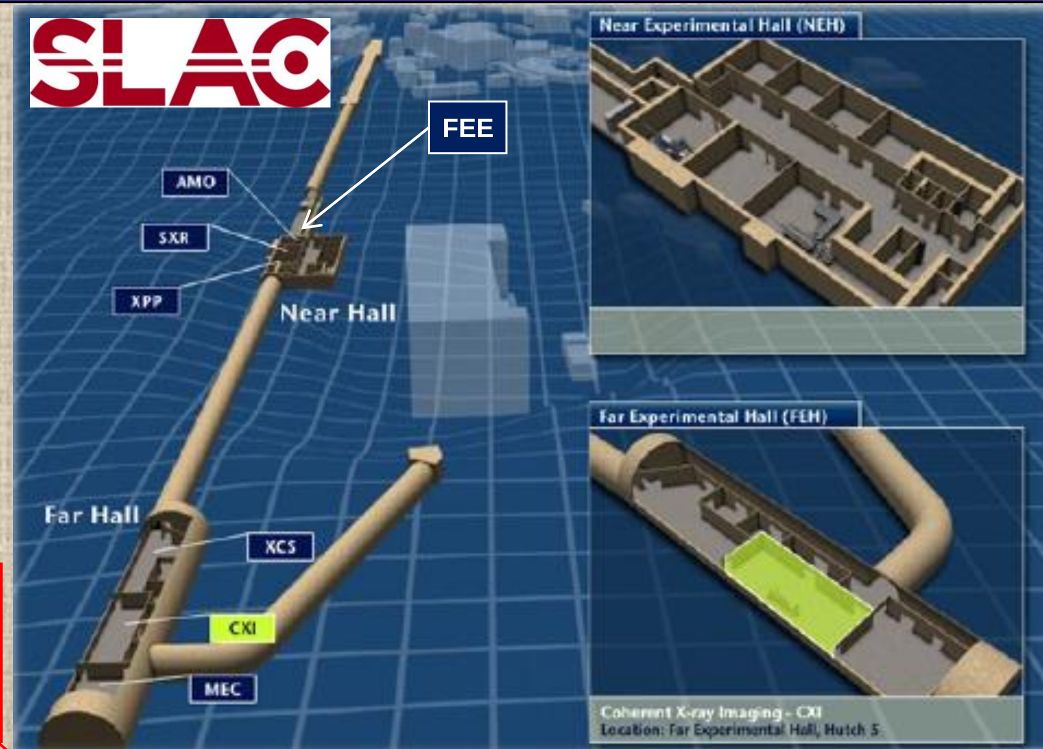
The Linac Coherent Light Source (LCLS)

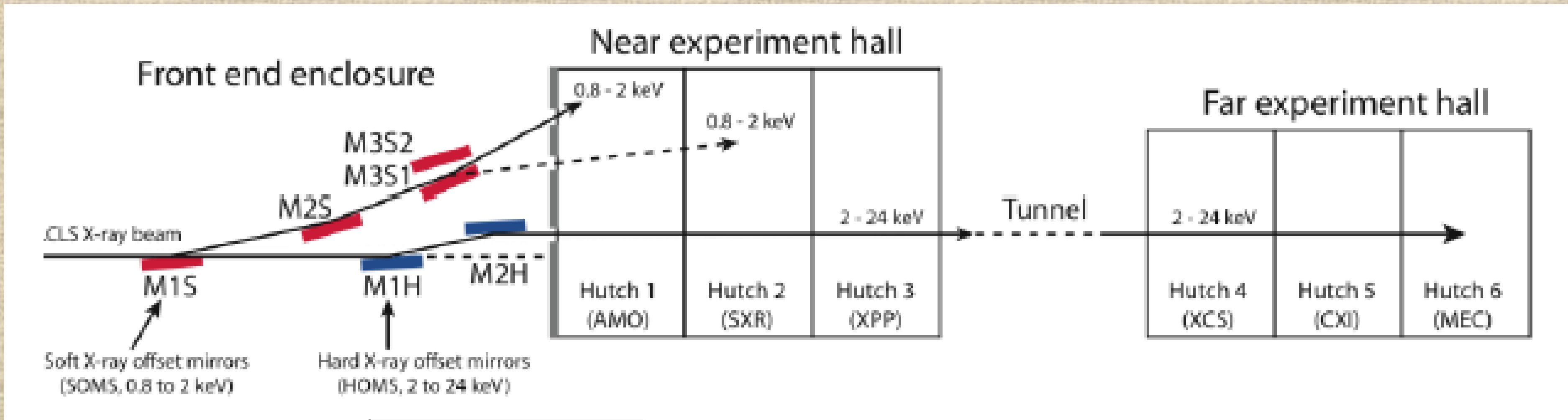
- **First x-ray free electron laser facility in the world.**
- **Extremely intense and short pulses of coherent x-rays.**
- **Spectral range from 0.5 to ~8 keV in the 1st harmonic, ~ 24 keV in the 3rd harmonic.**

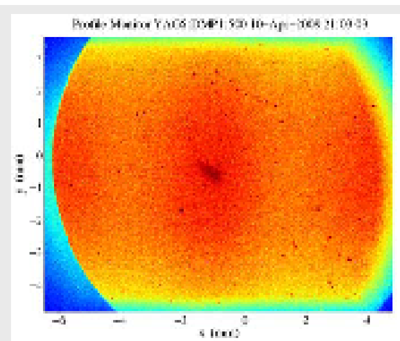


over 10 orders of magnitude higher than current 3rd generation synchrotron sources

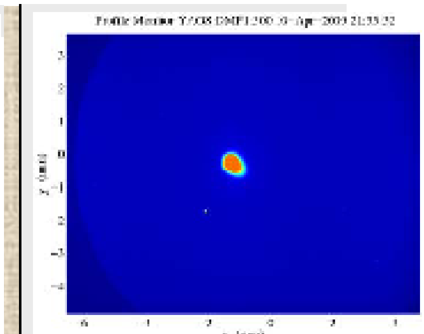
Parameter	0.827 keV	8.27 keV
FEL pulse (rms)	137 fs	73 fs
FEL width (FWHM)	81 μm	60 μm
FEL divergence (FWHM)	8.1 μrad	1.1 μrad
FEL peak brightness [photons s^{-1} mm^{-2} $mrad^{-2}$ $(0.1\%\ bw)^{-1}$]	0.28×10^{32}	15×10^{32}
FEL photons per pulse	20×10^{12}	2×10^{12}
Avg FEL power (at 120 Hz)	0.23 W	0.23 W

LLNL developed all (>20) mirrors and gratings for the LCLS





Spontaneous Beam



FEL beam

- **M. J. Pivovaroff et al., Proc. SPIE 6705, 67050O (2007).**
- **R. Soufli et al., Proc. SPIE 7077, 707716 (2008).**
- **T. J. McCarville et al., Proc. SPIE 7077, 70770E (2008).**
- **R. Soufli et al., Proc. SPIE 7361, 73610U (2009).**
- **A. Barty et al., Optics Express 17, 15508 (2009).**
- **R. Soufli, M. Fernandez-Perea et al., Appl. Opt. 51, 2118 (2012).**

Unique requirements for LCLS mirrors:

- **Withstand instantaneous peak power of LCLS FEL (materials)**
- **Coherence/intensity preservation of LCLS wavefront (< 2 nm rms figure, 0.25 nm rms MSFR)**
- **Pointing stability and resolution (< 1 μrad for soft x-ray offset mirrors, 100 nrad for hard x-ray offset mirrors)**

Soft x-ray region: 0.5 < $h\nu$ < 2 keV: B_4C coatings
Hard x-ray region: 2 < $h\nu$ < 8 (24.8) keV: SiC coatings

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X-ray mirror coating lifetime at LCLS

- 20 mirrors and gratings at LCLS, coated with B_4C and SiC
- C-based blemishes developed on 2 B_4C-coated KB focusing mirrors and on some of the offset mirrors. Other mirrors could be affected, including SiC-coated
- The C blemish development has been observed repeatedly in other EUV/soft x-ray installations. It is directly related to the beam impact combined with environmental conditions. It may be mitigated, but cannot be avoided (even in lowest and cleanest vacuum conditions)
- At some point the C blemish will degrade the coating optical performance to an unacceptable level
- A few re-alignment iterations will be possible before the entire clear aperture is fully degraded
- Our group at LLNL is involved in developing recovery techniques for LCLS mirrors

R. Soufli, M. Fernandez-Perea, et al., Proc. SPIE 8077 (2011)

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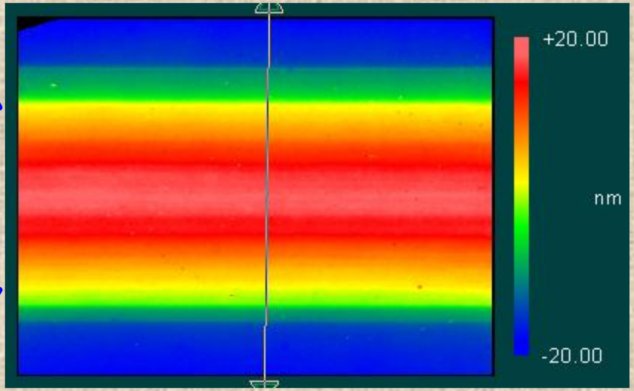
Coating blemish observation and characterization

• After ~ 9 months in operation, a line-shaped blemish was observed by visual inspection on one of the KB mirrors

Optical profilometry (Zygo NewView™), 2x magnification, at LLNL

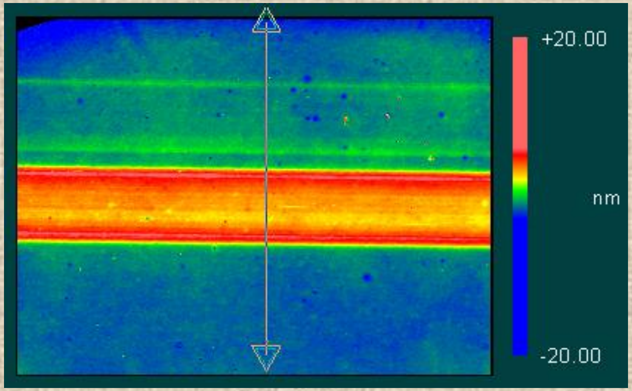
Main blemish



1.4 mm

2.95 mm

Satellite blemishes (not seen visually)



0.5 mm

2.95 mm

Note: Optical profilometry may provide inaccurate height information due to assumption of uniformity in the index of refraction.

• **Contact profilometry (Tencor P-11) at LLNL on a YAG witness piece confirmed that the blemish was indeed a deposit (i.e. positive height), with a height of 15 to 38 nm on the main stripe.**

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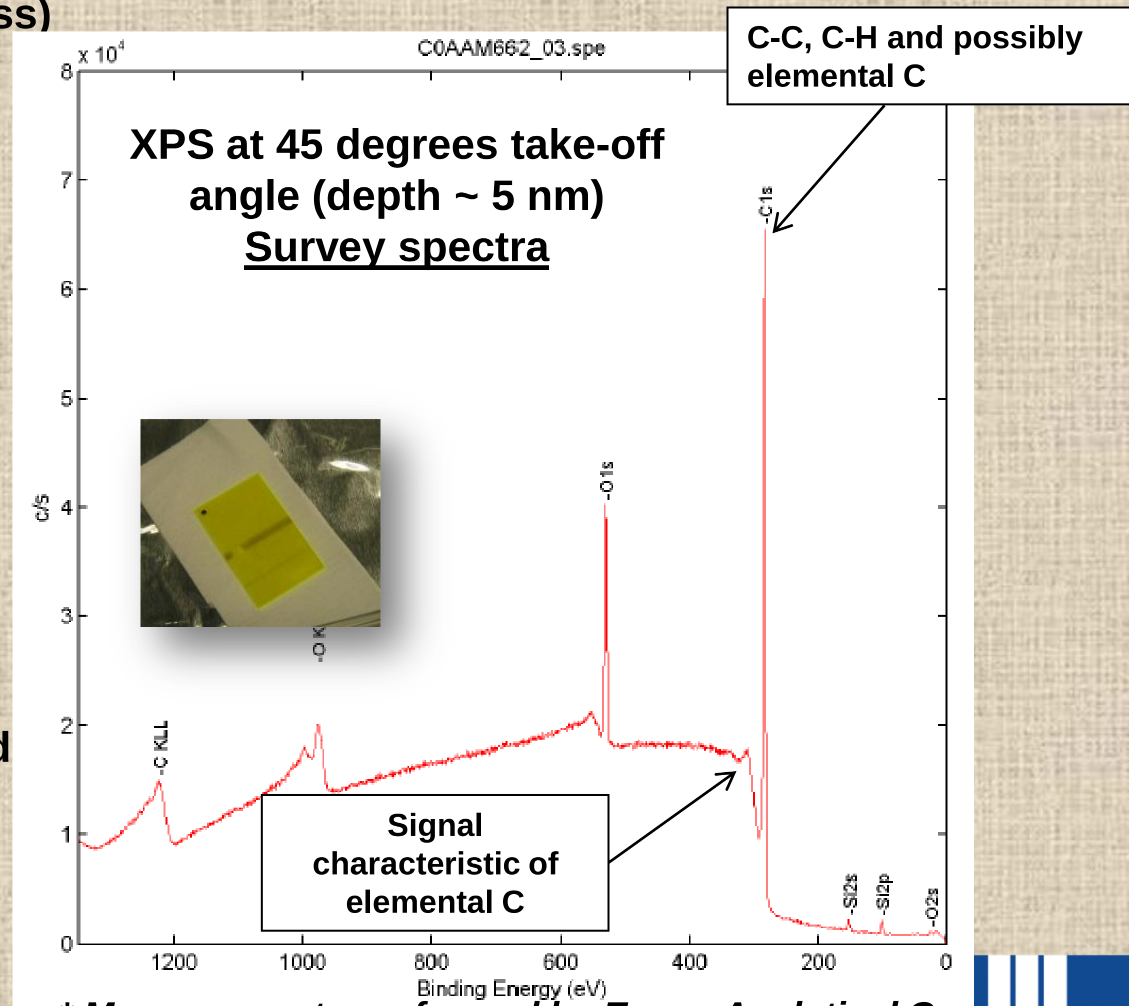
Composition analysis on blemish

- Rutherford Backscattering (RBS) analysis detected 100% carbon (across ~15 nm estimated thickness)

- X-ray photoelectron Spectroscopy (XPS) analysis on the blemish area showed atomic concentrations of 87% C, 12% O and 1% Si

- As for the chemical state, the blemish was composed primarily of carbon species, including hydrocarbon, O-containing organic species and possibly elemental carbon (e. g. graphite). Si was found as SiO_2.



** Measurements performed by Evans Analytical Group, Sunnyvale, CA*

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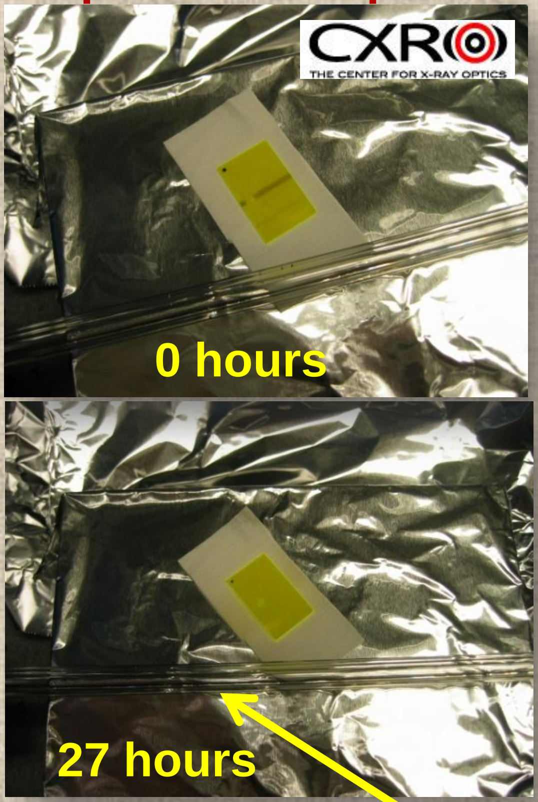
First attempts at UV-ozone cleaning on B_4C coatings

- **Dual low-pressure mercury UV lamp simultaneously produces and decomposes ozone to obtain oxygen atoms, which react with HCs to form volatile species (CO, CO_2, H_2O).**

- **Commercial UV lamps (ashers) are readily available**

- **The UV-ozone technique was applied first on the YAG witness piece with blemish from the AMO beamline. After 27 hours the C blemish was removed (as assessed by visual inspection)**

UV exposure set up at LBNL



Mercury lamp (λ ~184.9 and 253.7 nm)

- **Same exposure performed on a B_4C-coated (<u>no blemish</u>) Si wafer piece, with identical properties as the B_4C coatings on the LCLS KB mirrors**

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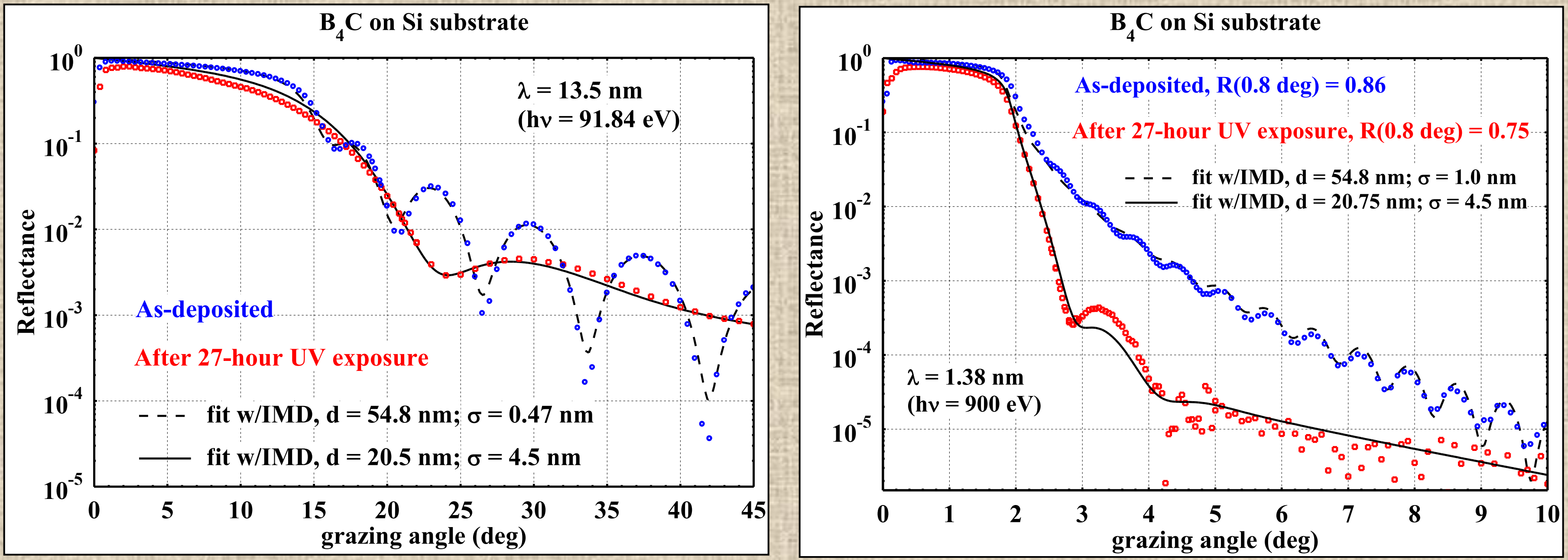
EUV and x-ray reflectance measurements and fits on B_4C-coated sample before/after UV-ozone exposure



Reflectance measurements performed at 6.3.2 beamline at ALS synchrotron, LBNL

- **The UV-ozone technique severely degrades the roughness, x-ray reflectance and thickness uniformity of the B_4C coating.**

- **Depth-sputtering XPS analysis (used in the fits) shows O-enriched top 3 nm, composed of B_2O_3 and B_4C and non uniformity across the film.**

Additional experiments are needed to understand the observed B_4C thickness decrease. Formation of volatile B- and C- compounds is a possibility

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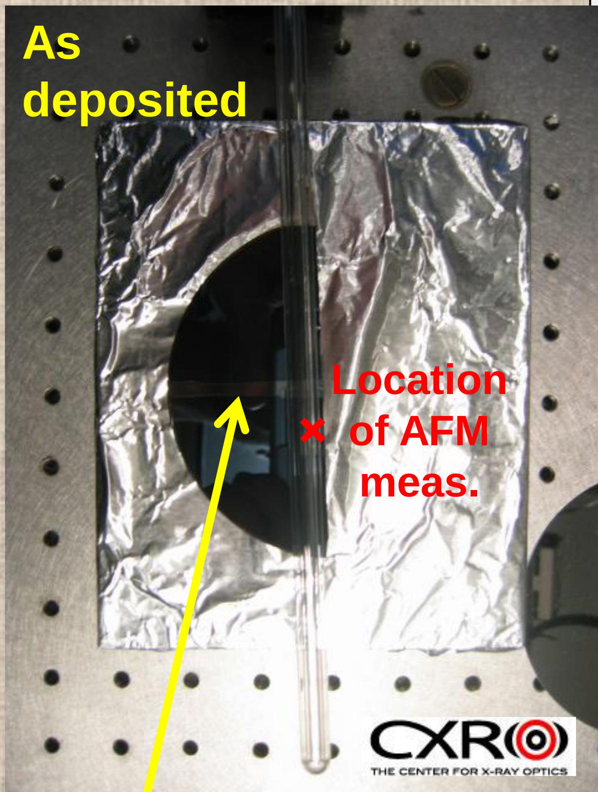
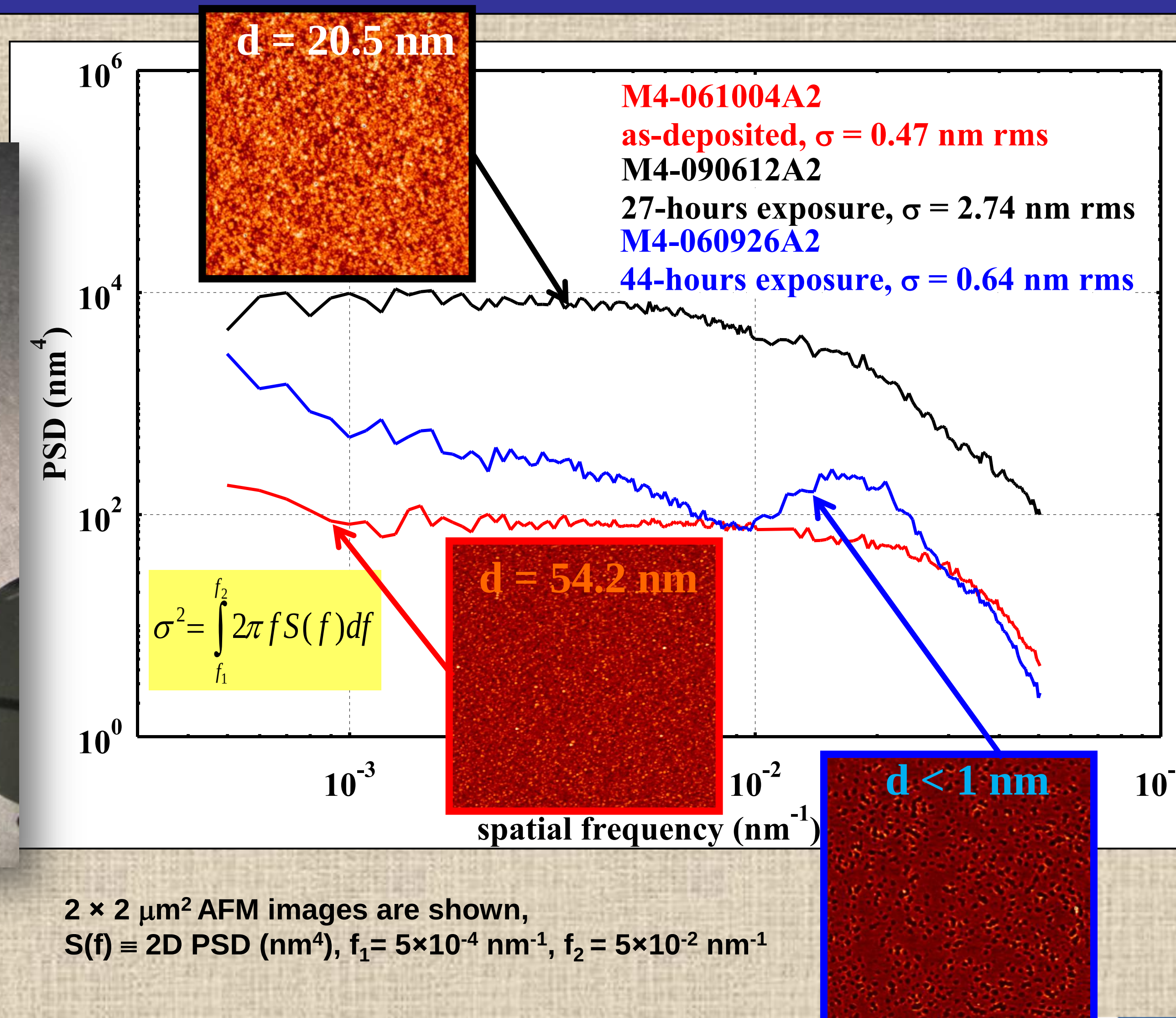
UV-ozone seems promising as removal technique for B_4C coatings

UV exposure set up at LBNL



Coating step (profilometer)



2 × 2 μm^2 AFM images are shown,
$S(f) \equiv$ 2D PSD (nm^4), $f_1 = 5\times10^{-4}\ nm^{-1}$, $f_2 = 5\times10^{-2}\ nm^{-1}$

Additional exposures, cleaning steps, and reflectance / composition measurements are planned to fully evaluate this possibility

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Implications for 6.x nm lithography mirrors

- Increased cost of 6.x nm-lithography mirrors due to very stringent specs makes their recovery even more desirable

- There may be several possible avenues to successfully recover 6.x nm-lithography mirrors (UV-ozone, plasma cleaning, release layers)

- We have first experimental results indicating that the UV-ozone technique successfully removes single B_4C layers. May also be applicable to single B layers, more experiments are needed

- Removal of B- and B_4C-based multilayers may involve additional removal techniques such as chemical or plasma etching

- Additional experiments should be planned also to optimize these techniques and ensure they are successful and scalable to full-size mirrors

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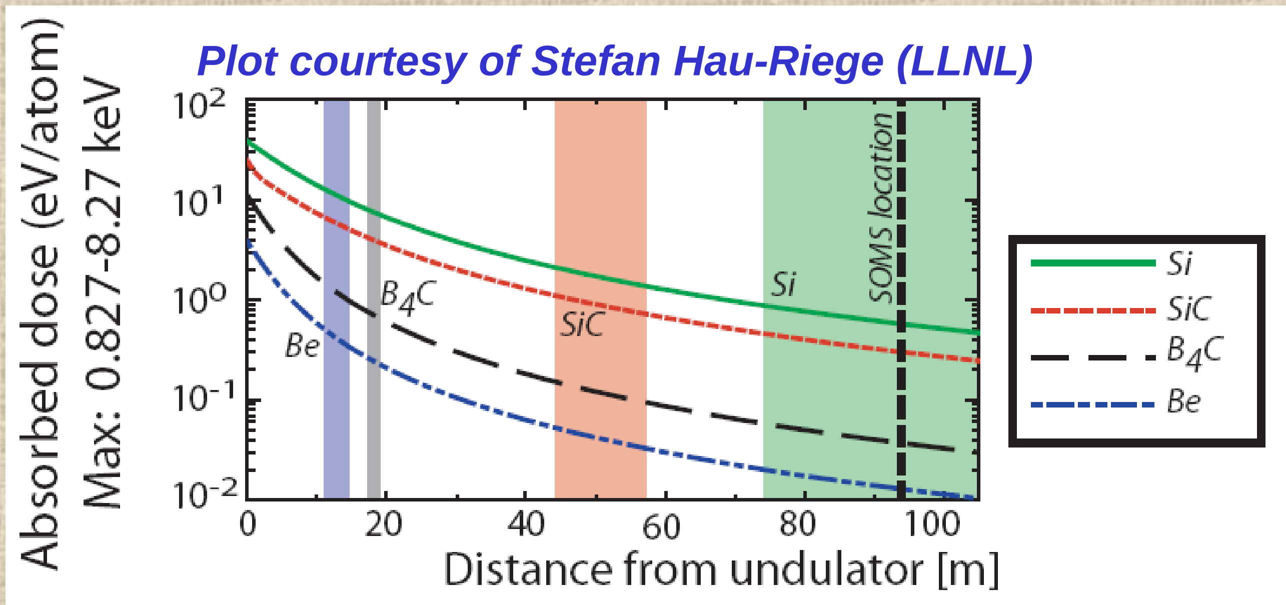
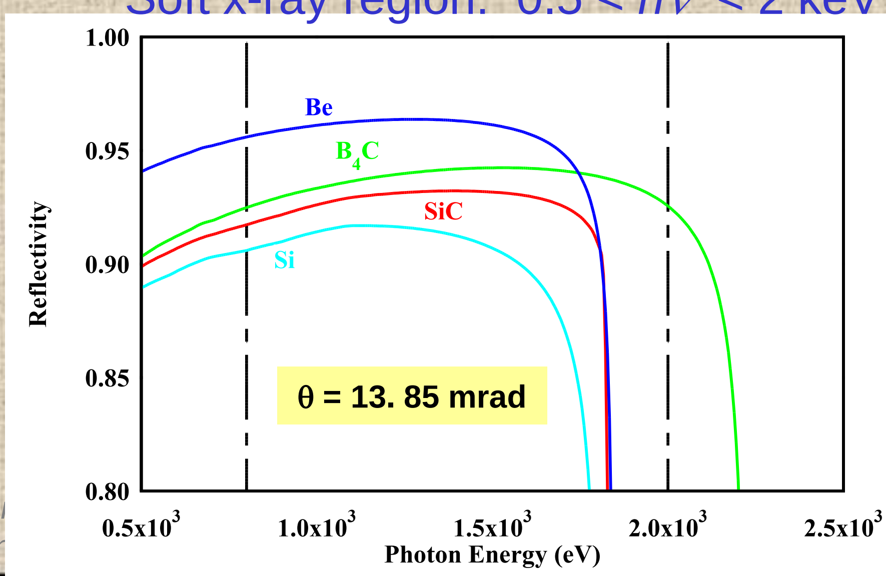
LCLS FEL beam dose considerations restrict the material choices

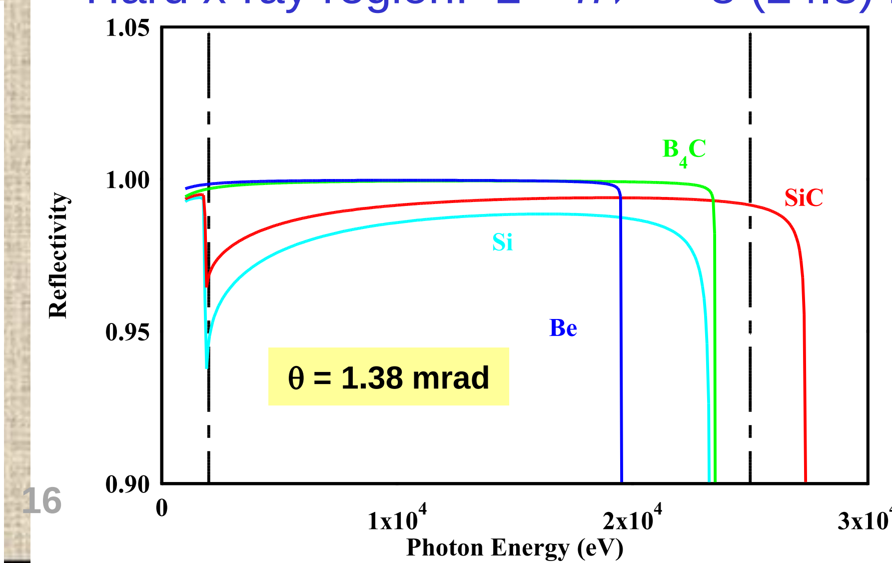


Vertical bands = range of distances over which the absorbed FEL dose will reach melting temperature, or melt each material

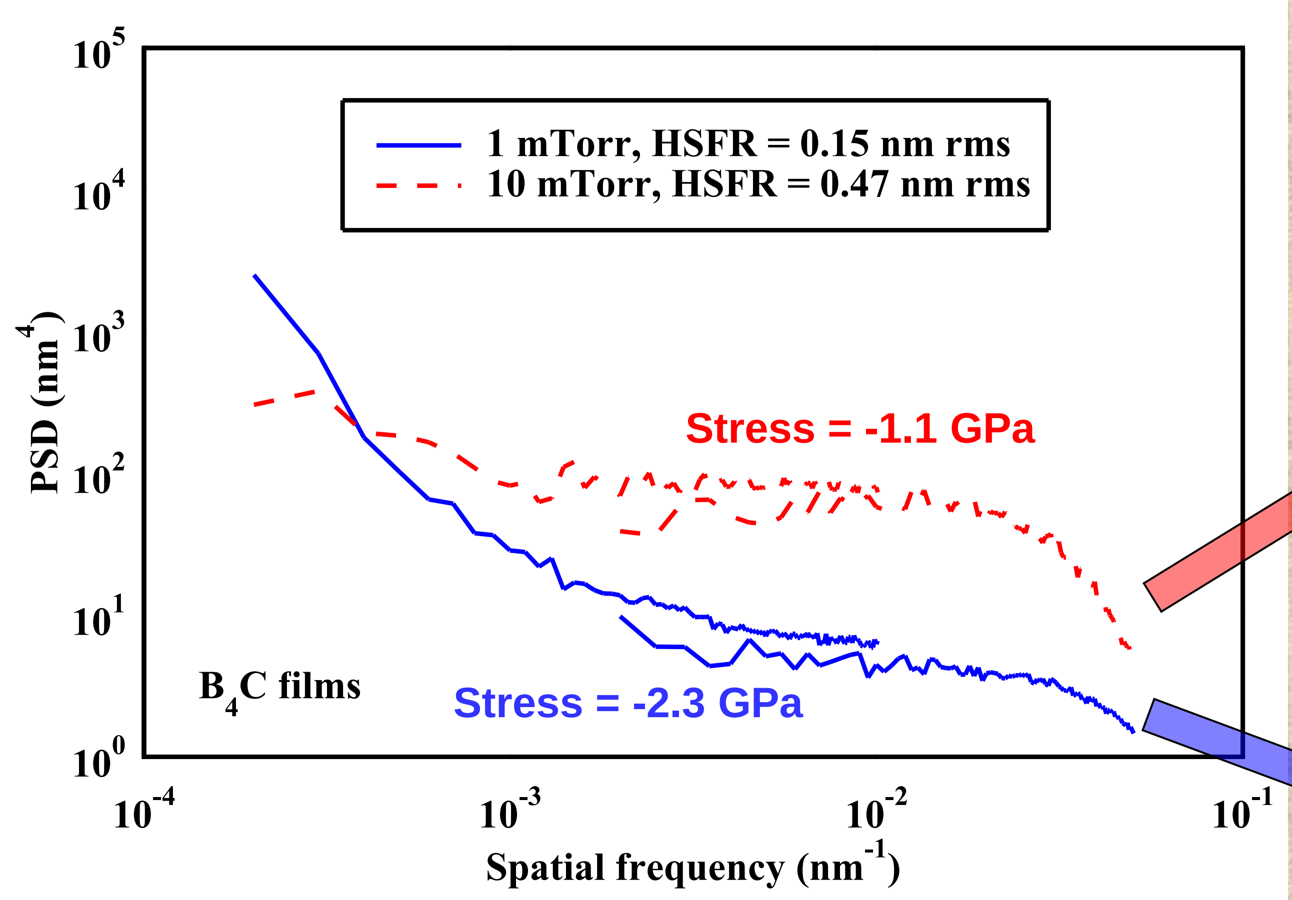
Soft x-ray region: $0.5 < h\nu < 2$ keV



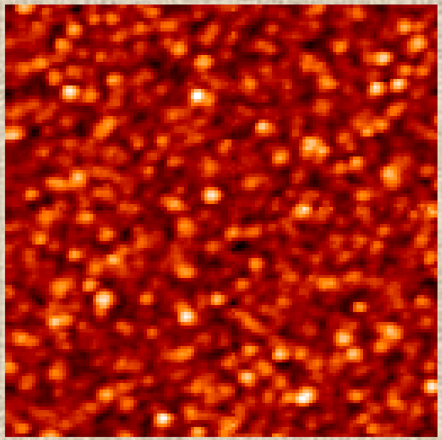
Hard x-ray region: $2 < h\nu < 8$ (24.8) keV



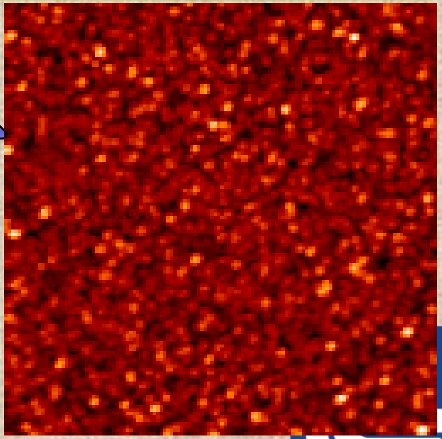
Boron carbide thin film development for LCLS SOMS mirrors



500×500 nm² detail from 2x2 μm² AFM scan



500×500 nm² detail from 2x2 μm² scan



$$\sigma^2 = 2\pi \int_{f_1}^{f_2} f\, S(f)\, df$$

where $S(f) \equiv$ PSD (nm^4), $f_1 = 5\times10^{-4}\,nm^{-1}$
$f_2 = 5\times10^{-2}\,nm^{-1}$

R. Soufli, S. L. Baker, J. C. Robinson, E. M. Gullikson, T. J. McCarville, M. J. Pivovaroff, S. P. Hau-Riege, R. M. Bionta, "Morphology, microstructure, stress and damage properties of thin film coatings for the LCLS x-ray mirrors", Proc. SPIE 7361, 73610U (2009).

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